

09/868211

*WOOL WITH ANTI FELT
AND METHOD FOR PROVIDING AN ANTI FELT
FINISH*

EPO/PTO 14 JUN 2001

Nonfelting wool and antifelt finishing process

The invention relates to nonfelting wool and to a process for antifelt finishing by treating the wool with a plasma, followed by an aftertreatment with various finishes.

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The textile processing industry has a particular interest in reducing the felting tendency of wool, especially of raw wool or unprocessed wool. The felting of wool is customarily reduced by finishing with specific auxiliaries.

- 10 Isocyanates for the antifelt finishing of textiles are well known and can be used, for example as described in DE-A-19 04 802, in organic solvents or, as described in DE-A-17 69 121, in aqueous dispersion with the aid of emulsifiers. Both organic solvents and possibly water-polluting emulsifiers are today no longer appropriate for ecological and occupational hygiene reasons. Prior artisans therefore developed self-dispersing isocyanates and also formulations containing very low levels of solvents or emulsifiers as auxiliaries and additives.
- 15
- 20 DE-A-1 794 221 describes the treatment of fibre materials with isocyanate prepolymers which still contain free isocyanate groups; this finishing process can take place in solvents such as perchloroethylene or in aqueous emulsion by using auxiliary emulsifiers.

US-A-3,847,543 discloses a process for the antifelt finishing of wool using an aqueous dispersion containing at one and the same time aliphatic isocyanates, OH-functional crosslinkers and organometallic catalysts. Although this process takes place in an aqueous phase, auxiliary solvents and emulsifiers continue to be required.

DE-A-26 57 513 describes a process for the antifelt finishing of wool by treating the wool yarn with an aqueous liquor which contains the feltproofing agent. The felt-proofing agents used are reactive polyolefins, reaction products of polyisocyanates and hydroxyl compounds, silicone polymers, aziridine compounds, reaction products of epoxides with fatty amines and dicarboxylic acids or polyamides, reaction products with thiosulphate end groups or preferably reaction products with mercapto end groups.

WO 95/30045 describes a process utilizing specific isocyanates for the antifelt finishing of wool. No solvents or emulsifiers are needed because the isocyanates used

"Express Mail" mailing label number: EPM168686445

Date of Deposit _____ June 14, 2001

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are water-dispersible. The wool is first subjected to a pretreatment with oxidizing agents, followed by a reductive treatment, before the water-dispersible isocyanates are used.

- 5 The prior art further includes another method for the antifelt finishing of wool where the wool is treated with a plasma. DE-A-43 44 428 discloses for example a process where the wool is subjected to an antifelt finish comprising a combination of plasma or corona pretreatment and enzymatic aftertreatment. The wool is sensitized with a solution which contains sulphide ions prior to the enzyme treatment.

- 10 DE 196 16 776 C1 further describes a process for the antifelt finishing of wool where moist wool material having a water content of 4-40% by weight is subjected to a low pressure plasma treatment before being further processed into textile fabrics or sheets. The wool is exposed to a radio frequency discharge of a frequency of 15 1 kHz-3 GHz and a power density of $0.001\text{-}3 \text{ W/cm}^3$ at a pressure of $10^{-2}\text{-}10 \text{ mbar}$ for a period of 1-600 sec in the presence or absence of non-polymerizing gases.

- The German Patent Application bearing the file reference 197 36 542.6, unpublished at the priority date of the present invention, discloses a process for the antifelt 20 finishing of wool where the wool is initially likewise exposed to a low pressure plasma and subsequently aftertreated with aqueous dispersions of self-dispersing isocyanates.

- All the aforementioned processes for the antifelt finishing of wool have the shared 25 disadvantage that applying the antifelt finishes has an adverse effect on the hand properties of the wool. The treated wool materials are frequently observed to have a strawy hand.

- The present invention accordingly has for its object to provide wool which is 30 nonfelting, i.e. felts or shrinks only insignificantly, if at all, in machine washing after further processing into made-up merchandise, and has improved hand properties.

- The present invention provides nonfelting wool, characterized in that the wool is
35 a) exposed to a plasma in a pretreatment,
b) optionally with an aqueous dispersion of self-dispersing isocyanates,
c) then with a softener and
d) finally optionally with an antislip agent.

Preferably the nonfelting wool is

- a) exposed to a plasma in a pretreatment,
- b) with an aqueous dispersion of self-dispersing isocyanates,
- 5 c) then with a softener and
- d) optionally finally with an antislip agent.

The present invention further provides the process for the antifelt finishing of wool, characterized in that the wool is

- 10 a) exposed to a plasma in a pretreatment,
 - b) optionally with an aqueous dispersion of self-dispersing isocyanates,
 - c) then with a softener and
 - d) finally optionally with an antislip agent.
- 15 Preference here is likewise given to a process for the antifelt finishing of wool that is characterized in that the wool is
 - a) exposed to a plasma in a pretreatment,
 - b) with an aqueous dispersion of self-dispersing isocyanates,
 - c) then with a softener and
- 20 d) optionally finally with an antislip agent.

The plasma treatment of the wool as per step a) in the process of the invention can be effected either in the form of a low temperature plasma treatment at reduced pressure or in the form of a corona treatment.

- 25 The wool used may be selected from a very wide range of wool materials, for example raw wool after the raw wool scour, dyed or undyed wool slubbing, dyed or undyed wool yarn, knits or cloths. The water content of the wool is customarily 4 40% by weight, preferably 5 to 30% by weight, particularly preferably 6 25% by weight, especially 8 15% by weight.

- 30 The low temperature plasma treatment is described at length in DE 196 16 776 C1, incorporated herein by reference. The wool is subjected to a high frequency discharge having a frequency of 1 kHz - 3 GHz and a power density of 0.001 - 3 W/cm³ at a pressure of 10⁻² - 10 mbar for a period of 1 - 600 seconds in the presence or absence of non-polymerizing gases.

The treatment is preferably effected under a pressure of 0.1 - 1 mbar and for a period of 2 - 5 minutes.

5 The actual low temperature plasma is generated by feeding in electromagnetic radiation in the frequency range of 1 kHz - 3 GHz. In a preferred variant, the low temperature plasma is generated via a microwave discharge of 1 - 3 GHz (the power density at the outcoupling is especially 0.1 - 15 W/cm²). The electromagnetic radiation can be supplied continuously or pulsed. A pulsed high frequency discharge having a pulsing frequency of up to 10 kHz is especially advantageous.

10 When non-polymerizing gases are additionally used as plasma process gases, they are introduced into the plasma treatment space at a flow rate of up to 200 l/h. Useful non-polymerizing gases are in particular oxygen, nitrogen, noble gases, especially argon, air or mixtures thereof.

15 The design and construction of a low temperature plasma reactor are known per se. Preference is given to using an electrodeless reactor having an outcoupling for microwaves. The wool to be treated is preferably placed underneath the outcoupling unit. The distance of the wool from the outcoupling unit is preferably 1 - 30 cm, especially 2 - 10 cm. After the wool to be treated has been introduced into the reactor, the reactor is suitably evacuated with vacuum pumps in such a way that the pressure during the plasma treatment is in the range of 10⁻² - 10 mbar, preferably 0.1 - 1 mbar. A continuous flow-through operation is preferably carried out by applying specific vacuum locks which make it possible for the material to enter and exit
25 without leakage.

30 Alternatively to this embodiment of the low temperature plasma treatment under low pressure, the wool can also be subjected to a corona treatment at a pressure in the range of 100 mbar - 1.5 bar, preferably at atmospheric pressure. The corona treatment is described in detail in a coterminously filed German patent application.

35 The corona treatment subjects the wool to a high frequency discharge having a power density of customarily 0.01-5 Ws/cm² for a period of 1-60 seconds, preferably 2-40 seconds, particularly 3-30 seconds, in the presence or absence of non-polymerizing gases. Suitable non-polymerizing gases are air, oxygen, nitrogen, noble gases or mixtures thereof.

The actual plasma is generated by applying an alternating voltage of 1-20 kV in the frequency range between 1 kHz-1 GHz, preferably 1-100 kHz, to electrodes, one or both poles being provided with an insulator material. The alternating voltage can be supplied either continuously or with individual pulses or with pulse trains and pauses in between.

- 5
- The design and apparatus configurations of a corona reactor are known per se and described for example in the German Application bearing the file reference 197 31 562, unpublished at the priority date of the present invention. The corona treatment is preferably carried out via electric discharges in the atmospheric pressure region, for which the wool to be treated is initially introduced into a sealed, tight treatment housing, charged there with the working gas, i.e. the abovementioned non-polymerizing gas, and subsequently exposed to an electric barrier discharge in a gap between the two treatment electrodes. The distance of the wool material from the treatment electrodes is 0-15 mm, preferably 0.1-5 mm, particularly 0.3-2 mm. The treatment electrodes are preferably constructed as rotatable rolls either or both of which are coated with electrically refractory dielectric material.
- 10
- 15
- Performing the corona treatment at a pressure in the range from 100 mbar to 1.5 bar, preferably at atmospheric pressure, has the advantage over the low pressure plasma treatment at 10^{-2} - 10 mbar that the equipment needed is very much less complicated than in the case of the low pressure treatment. Vacuum pumps are not required, nor is it necessary to fit special vacuum locks.
- 20
- 25
- 30
- The special effect of the plasma treatment in step a) of the process of the invention might be explained as follows. The liquid present in the fibre desorbs from the fibre surface as water vapour/gas during the process. High energy electrons, ions and also highly excited neutral molecules or radicals are formed and act on the surface of the fibre, the water vapour desorbed from the fibre ensuring that particularly reactive particles are formed in the immediate vicinity of the respective fibre surface and these particularly reactive particles act on the surface.

- Following the plasma treatment of wool in step a) an aftertreatment is carried out with various finishes. The facultative step b) comprises the treatment of the wool with an aqueous dispersion of self-dispersing isocyanates. Preferably step b) is carried out in the process of the invention. Useful self-dispersing isocyanates form part of the subject-matter of the German Patent Application bearing the reference
- 35

number 197 36 542.6, unpublished at the priority date of the present invention. They have an isocyanate content of 1-25% by weight, reckoned as NCO (having a molecular weight of 42 g/mol), and are obtainable by reaction in any order of

5 I) organic polyisocyanates having an average NCO functionality of 1.8 - 4.2
with

II) polyalkylene oxide alcohols, amines and/or thiols of the formula 1



where

15 n is 3-70,

X and Y are hydrogen or methyl with the proviso that when one of X and Y is methyl the other must be hydrogen,

20 R¹ and R² are independently straight-chain or branched C₁-C₆-alkyl radicals or straight-chain or branched C₁-C₆-acyl radicals, with the proviso that if R¹ is a straight-chain or branched C₁-C₆-acyl radical, R² can also be hydrogen, and, furthermore, R¹ and R² may also combine to form a -(CH₂)_m- alkylene radical where m = 4, 5, 6 or 7, wherein one or two CH₂ groups can be replaced by O and/or NH and/or one or two CH₂ groups can be substituted by methyl, and

25 Z is O, S or NH,

30 and optionally

III) further NCO-reactive compounds containing anionic, cationic and/or potentially anionic or cationic groups,

35 and optionally

IV) further auxiliary and additive substances.

For the purposes of the present invention, "self-dispersing" means that the isocyanates produce fine dispersions having particle sizes of <500 nm (measured by ultracentrifuge) in water when in a concentration of up to 70% by weight, preferably 5 up to 50% by weight.

Examples of useful starting materials for the self-dispersing isocyanates are:

10 I) Unmodified (i.e. not previously reacted with OH-functional compounds), aliphatic, cycloaliphatic, araliphatic or aromatic polyisocyanates having an average NCO functionality of 1.8 to 4.2. Preference is given to using aliphatic, cycloaliphatic, araliphatic or aromatic polyisocyanates which have uretdione and/or isocyanurate and/or allophanate and/or biuret and/or oxadiazine structures and which are preparable from aliphatic, cycloaliphatic, araliphatic or aromatic diisocyanates in a conventional manner.

15 Suitable examples of aliphatic and cycloaliphatic diisocyanates are 1,4-diisocyanatobutane, 1,6-diisocyanatohexane, 1,5-diisocyanato-2,2-dimethylpentane, 2,2,4- and 2,4,4-trimethyl-1,6-diisocyanatohexane, 1,3- and 1,4-diisocyanatocyclohexane, 1-isocyanato-3,3,5-trimethyl-5-isocyanatomethylcyclohexane, 1-isocyanato-1-methyl-4-isocyanatomethyl-cyclohexane and 4,4-diisocyanatodicyclohexylmethane or any mixtures of such diisocyanates.

20 Examples of suitable aromatic diisocyanates are tolylene diisocyanate, 1,5-diisocyanatonaphthalene and diphenylmethane diisocyanate.

25 The preferred polyisocyanates, which contain uretdione and/or isocyanurate and/or allophanate and/or biuret and/or oxadiazine groups and having an NCO content of 19 to 24% by weight which consist essentially of trimeric reaction products of 1,6-diisocyanatohexane or 1-isocyanato-3,3,5-trimethyl-5-isocyanatomethyl-cyclohexane and of the corresponding higher homologues.

30 Particular preference is given to using the corresponding polyisocyanates of the mentioned average NCO content which are substantially free of uretdione groups and have isocyanate groups and which are obtainable by conventional, catalytic trimerization of 1,6-diisocyanatohexane or 1-isocyanato-3,3,5-tri-

- 5 methyl-5-isocyanatomethyl-cyclohexane with isocyanurate formation and which preferably have an average NCO functionality of 3.2 to 4.2. Preference is also given to the trimeric polyisocyanates having an average NCO content of 19 to 24% by weight which are obtained in a conventional manner by reaction of 1,6-diisocyanatohexane with a deficiency of water or in the presence of water-eliminating reactants and which have essentially biuret groups.
- 10 II) Of the polyalkylene oxide alcohols, amines and/or thiols of the formula 1, the polyalkylene oxide alcohols are preferred ($Z = O$ in formula 1). The polyalkylene oxide alcohols can be reacted with NH_3 to form polyalkylene oxide amines ($Z = NH$ in formula 1) and with H_2S to form polyalkylene oxide thiols ($Z = S$ in formula 1).
- 15 The polyalkylene oxide alcohols thus underlying the polyalkylene oxide amines and thiols too contain on average 3-70, preferably 6-60, especially 7-20, alkylene oxide units per molecule and are obtainable in a conventional manner by alkoxylation of suitable starter molecules. The starter molecules used can be compounds of the formula R^1R^2NH . Depending on the meanings of R^1 and R^2 , they are secondary amines or amides. According to the definition of R^1 and R^2 mentioned for the formula 1, the alkoxylation reaction can also be started using morpholine as heterocyclic nitrogen compound. Identical compounds are further obtained on using compounds of the formula $R^1R^2N-CHX-CH-OH$, for example 2-morpholinoethanol, as starter molecules
- 20 25 for the alkoxylation reaction. Further useful starters include for example acylation products of ethanolamine, for example acetyl ethanolamine.
- 30 Alkylene oxides suitable for the alkoxylation reaction are in particular ethylene oxide and propylene oxide, which can be used in the alkoxylation reaction individually or in any desired order or else mixed. The polyalkylene oxide alcohols are in this case based either on pure polyethylene oxides or on mixed polyethylene oxides/propylene oxides. In particularly suitable polyalkylene oxide alcohols, there are on average 3-70, preferably 6-60 and in particular 7-20, alkylene oxide units per molecule and not less than 60 mol%, preferably not less than 70 mol%, of the alkylene oxide units are ethylene oxide units.

- III) The NCO-reactive compounds which contain anionic, cationic and/or potentially anionic or cationic groups are customarily
- 5 i) hydroxyl- or amino-functional compounds having tertiary amino groups as described in the German Patent Application DE-A-43 19 571, which is hereby expressly incorporated herein,
- 10 ii) hydroxyl- or amino-functional compounds having carboxyl or sulphonic acid groups as described in the German Patent Application DE-A-195 20 092, which is hereby expressly incorporated herein,
- 15 iii) hydroxyl- or amino-functional compounds having carboxylate or sulphonate groups whose counterions are metal cations of the alkali metal or alkaline earth metal group or ammonium ions, as likewise described in DE-A-195 20 092,
- 20 iv) hydroxyl- or amino-functional compounds having ammonium groups which are obtainable in a conventional manner from the tertiary amino groups of the compounds i) by alkylation or protonation as described in EP-A 0 582 166.
- 25 The process of the invention, as will be appreciated, may also be carried out using any desired mixtures of such NCO-reactive compounds, if chemically sensible, for example of the groups i) and iv) or of the groups ii) and iv).
- IV) The optional auxiliary and additive substances are for example wetting agents, surfactants, foam inhibitors or absorption assistants. These auxiliary and additive substances can either be inert or else reactive towards the isocyanate groups.
- 30 The unmodified polyisocyanates I to be used according to the invention can also be used in combination with external i.e. additional ionic or nonionic emulsifiers. Such emulsifiers are described for example in Methoden der organischen Chemie, Houben-Weyl, vol. XIV/1, part 1, page 190-208 Thieme-Verlag, Stuttgart (1961), in
- 35 US Patent 3,428,532 and EP-A 0,013,112. The emulsifiers are used in an amount sufficient to ensure dispersibility.

- If initially polyisocyanates I) are reacted with polyalkylene oxide alcohols II), this reaction can be carried out in a conventional manner, by maintaining an NCO/OH equivalents ratio of at least 2:1, generally of 4:1 to about 1000:1. Polyethylene oxide alcohols are used. To obtain polyethylene oxide-modified polyisocyanates having an
5 average NCO functionality of from 1.8 to 4.2, preferably of 2.0 to 4.0, containing 12.0 to 21.5% by weight of aliphatically or cycloaliphatically attached isocyanate groups and containing 2 to 20% by weight of ethylene oxide units (reckoned as C₂H₄O, molecular weight = 44 g/mol) within the polyethylene oxide chains, the polyethylene oxide chains having on average 3 to 70 ethylene oxide units.
10 The starting components I), II) and optionally III) can be reacted in any desired order in the absence of moisture, preferably without solvent. An increasing amount of component (II) will lead to a higher end-product viscosity. If the viscosity rises above 100 mPas, it is advantageous to carry out the process in the presence of a
15 solvent which is preferably miscible with water but inert towards the polyisocyanate. Suitable solvents are, for example, alkyl ether acetates, glycol diesters, toluene, carboxylic esters, acetone, methyl ethyl ketone, tetrahydrofuran and dimethylformamide.
20 Conventional catalysts such as dibutyltin dilaurate, tin(II) octoate or 1,4-diaza-bicyclo[2.2.2]octane in amounts of 10 to 1000 ppm, based on the components I), II) and optionally III), can be used to speed up the reaction of the components. The reaction is carried out in the temperature range up to 130°C, preferably in the range between 10°C and 100°C, particularly preferably between 20°C and 80°C. The
25 reaction is monitored by determining the NCO content by titration or by measurement of the IR spectra and evaluation of the NCO band at 2260-2275 cm⁻¹ and is terminated when the isocyanate content is not more than 0.1% by weight above the value which is obtained at complete conversion under the given stoichiometry. In general, reaction times of less than 24 hours are sufficient.
30 Preference is given to the solvent-free synthesis of the self-dispersing isocyanates to be used according to the invention.

In a further embodiment, it is also possible to prepare the self-dispersing isocyanates to be used according to the invention in step b) by mixing

- 35 1) unmodified polyisocyanates I)

- 2) polyisocyanates obtained by reaction of polyisocyanates I) with the NCO-reactive compounds III) at an equivalents ratio of the NCO-reactive groups of compounds III) to the NCO groups of component II, which are used, of 1:1 to 1:1000, and
- 5 3) polyisocyanates obtained by reaction of polyisocyanates I) with polyalkylene oxide alcohols, amines and/or thiols II), at an equivalents ratio of the NCO-reactive groups of component II to the NCO groups of component I), which are used, of 1:1 to 1:1000.

10 In this preparation variant, the person skilled in the art must make use of appropriate initial weights to control the number of the NCO-reactive equivalents, the polyalkylene oxide content, the NCO content and the NCO functionality in such a way that the mixture obtained has the composition required for water dispersibility, subject in particular to the preferred ranges already mentioned.

The self-dispersible isocyanates are industrially readily handleable and stable for many months in storage in the absence of moisture.

- 20 The self-dispersible isocyanates are preferably used without organic solvents in step b) of the process according to the invention. Due to their self-dispersibility, they are very easy to emulsify in water at temperatures up to 100°C without being subjected to high shearing forces. The isocyanate concentration of the emulsion can be up to 70% by weight. However, it is more advantageous to prepare emulsions
25 having an isocyanate concentration of up to 50% by weight. Emulsification may be accomplished using the mixing assemblies customary in the art (stirrers, mixers of the rotor-stator type and, for example, high pressure emulsifying machines). In general, a static mixer is sufficient. The emulsions obtained have a processing time of up to 24 hours, which depends on the structure of the self-dispersible isocyanates
30 used, in particular on their content of basic nitrogen atoms.

The treatment of the wool with the aqueous dispersion of the self-dispersing isocyanates in step b) is effected according to customary processes of the prior art. Suitable is for example a batchwise method by the exhaust process or a continuous
35 method by dipping, roll application, padding, application of a mist or spray or backwasher application optionally using dyeing machines, stirrers, etc. to agitate the

treatment liquor. The liquor ratio is choosable within wide limits and can be within the range of (20-5):1, preferably (10-5):1.

Step c) of the process according to the invention utilizes the following classes of substances as softeners: fatty acid amides, ester quats, quaternary fatty acid amides, betaines, fatty acid sarcosides, aminosilicones, polyethylene wax emulsions, silicone emulsions.

Step d) of the process according to the invention optionally treats the wool material with antislip agents. Antislip agents are finishes to prevent the mutual displacement of weft and warp threads in wovens and loop-formed knits produced later from the wool. There are antislip agents that will cause roughening of the fibre surface and antislip agents capable of dulling the fibre surface and of adhering the threads.

Useful antislip agents for roughening the fibre surface include in particular silica sols. These are aqueous solutions of approximately spherical, colloidally undissolved polysilicic acid molecules having an SiO₂ content of customarily 30 - 60%. Such silica sols are storable for years without undergoing any changes. Depending on the size of the particles, silica sol is milky cloudy to colourlessly clear. The average particle diameter is generally 5 to 150 nm. They are prepared in principle by treating aqueous alkali metal silicate solutions (waterglass) with ion exchangers and stabilizing with little alkali. Silica sols are commercially available in various surface modifications (anionic, cationic or nonionic).

Antislip agents that dull the fibre surface and adhere the threads are predominantly synthetic polymer dispersions and natural resins. Useful examples include synthetic polymer dispersions based on polyvinyl, polyacrylic, polymethacrylic, polystyrene or polybutadiene. Particular suitability is possessed by polyvinyl alcohols, polyacrylates and also blocked isocyanate resins and hydrophilicized isocyanate resins.

The application of the softeners in step c) and of the antislip agents in step d) of the process according to the invention is effected either batchwise in an exhaust process or continuously by dipping, roll application, padding, application of a mist or spray or backwasher application.

The isocyanate b) is used at 0.1-5% by weight, preferably at 0.5 - 2.5% by weight, based on the total weight of the liquor. The softener c) is used at 1 to 4% by weight,

preferably at 2-4% by weight, based on the total weight of the liquor. The antislip agent is used in an amount of 0.1 to 2% by weight, preferably 0.2 to 0.5% by weight, based on the total weight of the liquor.

- 5 In a further embodiment of the process according to the invention, the wool is
 - a) after the plasma pretreatment
 - b) treated with the aqueous dispersion of self-dispersing isocyanates and
 - c) finally with a softener.
- 10 The process of the invention thus includes in this case only the two aftertreatment steps b) and c) following step a). In a further variation of this embodiment, step a) can also be initially followed by the aftertreatment with the softener c) and subsequently with the treatment with the aqueous dispersion of self-dispersing isocyanates b). Another possibility is a conjoint treatment of the plasma treated wool
- 15 with the softener c) and the aqueous dispersion of self-dispersing isocyanates b).
- 20 In a further preferred embodiment, the process according to the invention includes all three aftertreatment steps b), c) and d) after the plasma pretreatment a). The order of the aftertreatments b), c) and d) of the wool material pretreated as per step a) can also be varied to the effect that the treatments b) and c) are carried out together and subsequently the aftertreatment with the antislip agent as per d) is executed. It is also possible to carry out the aftertreatment initially as per c), then as per b) and finally as per d). When an anionic or cationic silica sol is used as antislip agent d), the aftertreatment steps are preferably carried out in the order b), c), d) or c), b), d).

Examples

Percentages hereinbelow are by weight.

5 **B) Self-dispersing isocyanates**

B1) Self-dispersing isocyanate

85 parts by weight of an isocyanate having an NCO content of 22.5% and consisting essentially of trimeric hexamethylene diisocyanate are reacted at 60°C with 15 parts
10 by weight of an ethylene oxide polyether started on morpholine and having an average molecular weight of 420. The resultant product has an NCO content of 16.5% and a viscosity of 2550 mPas at 25°C. The product is very efficiently dispersible in a water-filled glass beaker simply by stirring with a glass rod. The arithmetic NCO functionality is F = 2.76.

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B2) Self-dispersing isocyanate

85 parts by weight of an isocyanate having an NCO content of 22.5% and consisting essentially of trimeric hexamethylene diisocyanate are reacted at 60°C with 15 parts by weight of a polyethylene glycol monomethyl ether having an average molecular
20 weight of 350. The resultant product has an NCO content of 17% and a viscosity of 1500 mPas at 25°C. The product is very efficiently dispersible in a water-filled glass beaker simply by stirring with a glass rod. The arithmetic NCO functionality is F = 2.7.

25 **C) Softeners**

C1) Softener

The softener C1 has the following composition:

10.3 parts by weight of polydimethylsiloxane
30 9.5 parts by weight of amide of myristic acid and ethylenediamine
4.0 parts by weight of a C₉-C₁₈-alcohol reacted with 3.6 mol of ethylene oxide
0.2 part by weight of isobutanol
76.0 parts by weight of water

35 **C2) Softener**

The softener C2 has the following composition:

- 2.3 parts by weight of amide of stearic acid and technical grade behenic acid with aminoethylethanolamine and N,N-dimethylamino-n-propylamine, quaternized with dimethyl sulphate
3.2 parts by weight of polydimethylsiloxane
5 17.0 parts by weight of amide of technical grade behenic acid and triethylene-tetramine acidified with acetic acid
12.0 parts by weight of cationically emulsified polyethylene oxide wax
20.9 parts by weight of fatty acid amide with nonionic emulsifier
44.6 parts by weight of water

10

C3) Softener

The softener C3 has the following composition:

- 80.0 parts by weight of nonionic polyethylene oxide wax emulsion
5.0 parts by weight of polyether siloxane
15 1.4 parts by weight of a salt of dibutyl phosphate and diethanolamine
1.0 part by weight of a C₁₂₋₁₃-fatty alcohol reacted with 10 mol of ethylene oxide
12.0 parts by weight of water
0.6 part by weight of scent

20

D) Antislip agents

D1) Antislip agent

The antislip agent D1 is a reaction product of:

- 85 parts by weight of a technical grade isocyanate from the trimerization reaction
25 of hexamethylene diisocyanate with
15 parts by weight of a technical grade polyethylene glycol monomethyl ether having a molecular weight of 350.

D2) Antislip agent

- 30 The antislip agent D2 is a mixture of
30 parts by weight of a silica sol cationized with basic aluminium chloride and stabilized with acetic acid and
70 parts by weight of water.

35 Examples 1-4

(Order of treatment steps: a), c), b))

- First, moist wool slubbing is subjected to a corona treatment (treatment conditions: frequency: 23 kHz; power: 3.7 kW; electrode distance: 0.8 mm; forward feed rate: 10 m/min) and thereafter in a waterbath initially to a treatment with a 2% aqueous solution of the particular softener. The wet slubbing is then whizzed to spin off excess softener solution. After air drying, the thusly pretreated wool slubbing is treated on a lab pad-mangle with an aqueous dispersion of the particular isocyanate (concentration of the isocyanate: 25 g/l of buffer solution (sodium acetate/glacial acetic acid buffer) to a wet pick-up of 80%. After rinsing out with water and air drying, the slubbing is tested by the Aachen felting ball test (IWTO standard 20-69) for the quality of the antifelt finish. The larger the felting ball diameter and the smaller the felt density, the better the antifelt finish. In addition, 4 people compare the hand of these wool slubbing samples independently to determine the hand ranking (ratings from 1 (best hand) to 12 (worst hand)).
- 15 Table (I) below contains the observed values for the felting ball diameter, the felt density and the hand ranking.

Table (I):

Example	1	2	3	4
Self-dispersing isocyanate	B2	B1	B2	B1
Softener	C1	C1	C2	C2
Felting ball diameter [cm]	2.282	2.246	3.019	2.869
Felt density [g/cm ³]	0.161	0.169	0.069	0.081
Hand ranking	12	9	10	7

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Examples 5-8

(Order of treatment steps: a), b), c))

- First, moist wool slubbing is subjected to a plasma treatment similarly to Examples 25 1-4 and then treated on a lab pad-mangle with an aqueous dispersion of the particular self-dispersing isocyanate (concentration of the isocyanate: 25 g/l of buffer solution (sodium acetate/glacial acetic acid buffer) to a wet pick-up of 80%. After air drying, the thusly pretreated wool slubbing is treated in a waterbath with a 2% aqueous solution of the particular softener. The wet slubbing is then whizzed to spin off excess softener solution. After rinsing out with water and air drying, the slubbing is

tested by the Aachen felting ball test (IWTO standard 20-69) for the quality of the antifelt finish. In addition, 4 people compare the hand of these wool slubbing samples independently to determine the hand ranking.

- 5 Table (II) below contains the observed values for the felting ball diameter, the felt density and the hand ranking.

Table (II):

Example	5	6	7	8
Self-dispersing isocyanate	B2	B1	B2	B1
Softener	C1	C1	C2	C2
Felting ball diameter [cm]	2.842	3.189	3.181	2.681
Felt density [g/cm ³]	0.083	0.059	0.059	0.099
Hand ranking	2	4	6	1

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Examples 9-12:

(Order of treatment steps: a), b), c), d))

- 15 First, moist wool slubbing is subjected to a plasma treatment as described in Examples 1-4 and then treated on a lab pad-mangle with an aqueous dispersion of the particular self-dispersing isocyanate (concentration of the isocynate: 25g/e of sulfer solution (sodium acetate/glacial acetic acid sulfer)) to a wet pick-up of 80%. After rinsing out with water and air drying the thusly pretreated wool slubbing is first treated in a waterbath with a 2% aqueous solution of the particular softener. The wet slubbing is then whizzed to spin off excess softener solution. After air drying, the slubbing is (likewise in a waterbath) treated with a 1% aqueous solution of the antislip agent. After squeezing off and complete air drying, the slubbing is tested by the Aachen felting ball test (IWTO standard 20-69) for the quality of the antifelt finish. In addition, 4 people compare the hand of these wool slubbing samples independently to determine the hand ranking.
- 20
- 25

Table (III) below contains the observed values for the felting ball diameter, the felt density and the hand ranking.

Table (III):

Example	9	10	11	12
Self-dispersing isocyanate	B2	B1	B2	B1
Softener	C1	C1	C2	C2
Antislip agent	D1	D1	D1	D1
Felting ball diameter [cm]	3.298	2.610	3.274	2.825
Felt density [g/cm ³]	0.053	0.107	0.054	0.085
Hand ranking	8	5	11	3

Examples 13-16

- 5 (Order of treatment steps: a), b), c), d));
Comparison of various antislip agents
- Similarly to Examples 1-12, wool slubbing is first subjected to a corona treatment and then treated with an aqueous liquor containing 10 g/l of the water-dispersible
10 isocyanate B2 and squeezed off on a lab pad-mangle to a wet pick-up of 80%. After rinsing out with water and air drying, the thusly pretreated wool slubbing is initially subjected in a waterbath and an exhaust process to treatment with a 2% aqueous solution of the particular softener. The wet slubbing is then whizzed to spin off excess softener solution. After air drying, the slubbing is, likewise in a waterbath,
15 treated with an antislip agent (concentration of the antislip agent: 1% in water) in an exhaust process. After squeezing off, rinsing out and complete air drying, the slubbing is tested for the quality of its antifelt finish by the Aachen felting ball test (IWTO standard 20-69).
- 20 Table (IV) below shows the values observed for the felting ball diameter and the felt density.

Table (IV)

Example	13	14	15	16
Self-dispersing isocyanate	B2	B2	B2	B2
Softener	C1	C3	C1	C3
Antislip agent	D2	D2	D1	D1
Felting ball diameter [cm]	3.439	3.742	3.619	3.796
Felt density [g/cm ³]	0.047	0.037	0.040	0.035

DISPERSANT = DEDICATE